



March 29, 2016

Tom Moe USS Corporation P.O. Box 417 Mountain Iron, MN 55768

RE: Project: NPDES-TB Wk3 Pace Project No.: 1262656

#### Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on March 16, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Melisa M Woods

Mazzi Wirds

melisa.woods@pacelabs.com

Project Manager

**Enclosures** 

cc: Terri Sabetti, NTS





315 Chestnut Street Virginia, MN 55792 (218) 742-1042



### **CERTIFICATIONS**

Project: NPDES-TB Wk3

Pace Project No.: 1262656

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792 Alaska Certification #MN01084 Arizona Department of Health Certification #AZ0785 Minnesota Dept of Health Certification #: 027-137-445

Wisconsin DNR Certification #: 998027470 WA Department of Ecology Lab ID# C1007

Nevada DNR #MN010842015-1 Oklahoma Department of Environmental Quality

North Dakota Certification: # R-203

**Duluth Minnesota Cerification ID's** 

4730 Oneota St., Duluth, MN 55807 Minnesota Dept of Health Certification #: 027-137-152 Wisconsin DNR Certification #: 999446800

North Dakota Certification #: R-105





# **SAMPLE SUMMARY**

Project: NPDES-TB Wk3

Pace Project No.: 1262656

| Lab ID     | Sample ID         | Matrix | Date Collected | Date Received  |
|------------|-------------------|--------|----------------|----------------|
| 1262656001 | SD 001 (Seep 020) | Water  | 03/16/16 11:15 | 03/16/16 14:05 |





# **SAMPLE ANALYTE COUNT**

Project: NPDES-TB Wk3

Pace Project No.: 1262656

| Lab ID     | Sample ID         | Method       | Analysts | Analytes<br>Reported | Laboratory |
|------------|-------------------|--------------|----------|----------------------|------------|
| 1262656001 | SD 001 (Seep 020) | EPA 1664 TPH | BT1      | 1                    | PASI-DUL   |
|            |                   | USGS I-3765  | JP1      | 1                    | PASI-V     |



# **ANALYTICAL RESULTS**

Project: NPDES-TB Wk3

Pace Project No.: 1262656

Date: 03/29/2016 04:20 PM

| Sample: SD 001 (Seep 020)    | Lab ID:    | 1262656001  | Collected: 03/16/16 11:15 |      |    | Received: 03 | /16/16 14:05 Ma | atrix: Water |      |
|------------------------------|------------|-------------|---------------------------|------|----|--------------|-----------------|--------------|------|
| Parameters                   | Results    | Units       | Report<br>Limit           | MDL  | DF | Prepared     | Analyzed        | CAS No.      | Qual |
| 1664 SGT-HEM, TPH            | Analytical | Method: EPA | 1664 TPH                  |      |    |              |                 |              |      |
| Total Petroleum Hydrocarbons | ND         | mg/L        | 3.1                       | 0.94 | 1  |              | 03/28/16 17:21  |              | L2   |
| USGS I-3765 TSS              | Analytical | Method: USG | S I-3765                  |      |    |              |                 |              |      |
| Total Suspended Solids       | 1.2        | mg/L        | 1.0                       | 1.0  | 1  |              | 03/17/16 12:35  |              |      |



#### **QUALITY CONTROL DATA**

Project: NPDES-TB Wk3

Pace Project No.: 1262656

QC Batch: DUL/5695 Analysis Method:
QC Batch Method: EPA 1664 TPH Analysis Description:

Analysis Description: 1664 SGT-HEM, TPH

**EPA 1664 TPH** 

Associated Lab Samples: 1262656001

METHOD BLANK: 300567 Matrix: Water

Associated Lab Samples: 1262656001

Blank Reporting
Parameter Units Result Limit MDL Analyzed Qualifiers

Total Petroleum Hydrocarbons mg/L ND 3.0 0.90 03/28/16 15:42

LABORATORY CONTROL SAMPLE: 300568

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers 64-132 L0 Total Petroleum Hydrocarbons mg/L 20 10.2 51

MATRIX SPIKE SAMPLE: 300569

Date: 03/29/2016 04:20 PM

MS MS 1262733001 Spike % Rec Parameter Units Result Conc. Result % Rec Limits Qualifiers 1.2J 64-132 Total Petroleum Hydrocarbons 20.8 15.9 71 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



#### **QUALITY CONTROL DATA**

USGS I-3765

Project: NPDES-TB Wk3

Pace Project No.: 1262656

QC Batch: WET/22993 Analysis Method:

QC Batch Method: USGS I-3765 Analysis Description: USGS I-3765 Total Suspended Solids

Associated Lab Samples: 1262656001

METHOD BLANK: 297742 Matrix: Water

Associated Lab Samples: 1262656001

Blank Reporting
Parameter Units Result Limit MDL Analyzed Qualifiers

Total Suspended Solids mg/L ND 1.0 03/17/16 12:33

LABORATORY CONTROL SAMPLE: 297743

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers **Total Suspended Solids** mg/L 239 210 88 80-120

SAMPLE DUPLICATE: 297744

1262597001 Dup Max **RPD RPD** Parameter Units Result Result Qualifiers 270 10 Total Suspended Solids 245 10 mg/L

SAMPLE DUPLICATE: 297745

Date: 03/29/2016 04:20 PM

1262591001 Dup Max RPD RPD Parameter Units Result Result Qualifiers 85.0 Total Suspended Solids mg/L 82.5 3 10

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



#### **QUALIFIERS**

Project: NPDES-TB Wk3
Pace Project No.: 1262656

#### **DEFINITIONS**

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

**DUP - Sample Duplicate** 

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

#### **LABORATORIES**

PASI-DUL Pace Analytical Services - Duluth
PASI-V Pace Analytical Services - Virginia

#### **ANALYTE QUALIFIERS**

Date: 03/29/2016 04:20 PM

- LO Analyte recovery in the laboratory control sample (LCS) was outside QC limits.
- L2 Analyte recovery in the laboratory control sample (LCS) was below QC limits. Results may be biased low.



# **QUALITY CONTROL DATA CROSS REFERENCE TABLE**

Project: NPDES-TB Wk3

Pace Project No.: 1262656

Date: 03/29/2016 04:20 PM

| Lab ID     | Sample ID         | QC Batch Method | QC Batch  | Analytical Method | Analytical<br>Batch |
|------------|-------------------|-----------------|-----------|-------------------|---------------------|
| 1262656001 | SD 001 (Seep 020) | EPA 1664 TPH    | DUL/5695  |                   |                     |
| 1262656001 | SD 001 (Seep 020) | USGS I-3765     | WET/22993 |                   |                     |

Mt. Iron, MN 55768 Address: Company:

Required Client Information:

USS Corporation P.O. Box 417

Requested Due Date:

Fax

Purchase Order #:
Project Name: Project #:

NPDES-TB Wk3

Copy To:

ITEM #

Sample ids must be unique One Character per box. (A-Z, 0-9/, -) SAMPLE ID

MATTRIX
Drinking Water
Water
Waste Water
Waste Water
Product
Soil/Soild
Oil
Wipe
Air
Other
Tissue

MATRIX CODE (see valid codes to left)

START

8

(G=GRAB C=COMP)

COLLECTED

SAMPLE TYPE

3-16761115

DATE

拱

DATE

TIME

SD 001 (Seep 020)

Report To: Tom Moe Required Project Information: The Chain-of-Custody is a LEGAL DOCUMEN WO#: 1262656 CHAIN-OF-CUSTODY / Analyting Attention: Company Name: Invoice Information: CLIENT: USS CORP PM: MMW

Page 10 of 13

SIGNATURE of SAMPLER: PRINT Name of SAMPLER: 3-676 11:15 3-16-76 SAMPLE TEMP AT COLLECTION 14,08 # OF CONTAINERS Pace Profile #: Pace Project Manager: Address: Unpreserved law proteila H2SO4 HNO3 Preservatives HCI NaOH heather.zika@pacelabs.com, Na2S2O3 Methanol Other Analy content Yart TSS DATE Signed: TRPH 1664 Due Date: 03/30/16 3-16-1 14:05 TEMP in C Residual Chlorine (Y/N) Received on lce (Y/N) Custody Sealed ð Cooler ح (Y/N) Samples Intact (Y/N)

Sand noute

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3-16-16



**FECAL WAIVER ON FILE** 

Document Name:

Sample Condition Upon Receipt Form

Document No.: F-VM-C-001-Rev.09 Document Revised: 23Feb2015 Page 1 of 1

Issuing Authority:

Pace Virginia, Minnesota Quality Office WO#:1262656 Sample Condition **Client Name:** Project #: **Upon Receipt** Courier: Fed Ex USPS Client Commercial □ Pace Other: **Tracking Number:** Custody Seal on Cooler/Box Present? Optional: Proj. Due Date: Yes ☑No Proj. Name: Seals Intact? □Yes A TNo Bubble Bags Packing Material: Bubble Wrap ₩one Other: Temp Blank? □No Thermometer Used: 140792808 ₩et □Blue None Samples on ice, cooling process has begun Cooler Temp Read °C: Cooler Temp Corrected °C: ☐Yes **Biological Tissue Frozen?** Temp should be above freezing to 6°C Correction Factor: 0,3 Date and Initials of Person Examining Contents: Comments: Chain of Custody Present? **Z**Yes □No □N/A Chain of Custody Filled Out? **[**Z]Yes □No □N/A 2. Chain of Custody Relinquished? **1**Yes □No □N/A 3. Sampler Name and Signature on COC? ☑Yes □No □N/A 4. Samples Arrived within Hold Time? **⊠**Yes □No □N/A Short Hold Time Analysis (<72 hr)? Yes □No □N/A 6. Rush Turn Around Time Requested? □Yes ☑No □N/A 7. Sufficient Volume? **☑**Yes □No □N/A 8. Correct Containers Used? [Z]Yes □No □N/A 9. -Pace Containers Used? ✓Yes □No □N/A Containers Intact? ZÎŶes □No □N/A Filtered Volume Received for Dissolved Tests? ∏уе<u>s</u> □No ✓N/A 11. Note if sediment is visible in the dissolved containers. Sample Labels Match COC? □Yes □No □N/A -includes Date/Time/ID/Analysis Matrix: See pH log for results and additional preservation All containers needing acid/base preservation will be □Yes □No ☑N/A checked and documented in the pH logbook. documentation Headspace in Methyl Mercury Container Yes □No ØN/A 13. Headspace in VOA Vials (>6mm)? Yes □No ☑N/A 14. Trip Blank Present? □Yes ПNо ĎN/A 15. Trip Blank Custody Seals Present? Yes □No **□**N/A Pace Trip Blank Lot # (if purchased): CLIENT NOTIFICATION/RESOLUTION Field Data Required? Yes No Person Contacted: Date/Time: Comments/Resolution:

Project Manager Review: 4 Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office ( i.e out of hold, Incorrect preservative, out of temp, incorrect containers)

**TEMPERATURE WAIVER ON FILE** 

# Intra-Regional Chain of Custody



| 3                             | 4        | ယ | 2  | -                        | Transfers         |             | Ċħ | 4 | ω | 2 | 1                 | ltem.                |                      | Pace<br>315<br>Virgi  | Rece   | 440   |
|-------------------------------|----------|---|----|--------------------------|-------------------|-------------|----|---|---|---|-------------------|----------------------|----------------------|---|--|---|
| Cooler Temperature on Receipt |          |   | W. |                          | sfers Released By |             |    |   |   |   | SD 001 (Seep 020) | Sample ID            |                      | Pace Analytical Virginia<br>315 Chestnut Street<br>Virginia, MN 55792<br>Phone (218) 742-1042 | Received at:   | WOINGIGHT 1202000   |
| -                             |          |   |    |                          |                   |             |    |   |   |   | PS                | Sample<br>Type       |                      |   |  | TOTAL CONTROL OF THE PROPERTY |
| ر<br>ا                        |          |   |    | 211/16                   | Date∕Time         |             |    |   |   |   | 3/16/2016 11:15   | Collect<br>Date/Time |                      | Pace Analytical Dulu<br>4730 Oneota Street<br>Duluth, MN 55807<br>Phone (218) 727-63          | Send To Lab:   |   |
| Custody Soal ( v)             |          |   |    | 716Wellow Atm a Polasson | Received By       |             |    |   |   |   | 1262656001        | Lab ID               |                      | Pace Analytical Duluth<br>4730 Oneota Street<br>Duluth, MN 55807<br>Phone (218) 727-6380      |  |   |
| )<br>2. Z                     |          |   |    | a Poppar                 | ,                 |             |    |   |   |   | Water 2           | Matrix E             | ्. Pře               |   | And the second of the second o |   |
| Deceived on                   |          |   |    |                          | Da                |             |    |   |   |   |                   |                      | Preserved Containers |   |  |   |
| A Joseph                      | )<br>    |   | i  | 少て<br><u> </u>           | Date/Time         |             |    |   |   |   | ×                 | E                    | PA 1664 TPH          |   |  |   |
| ,<br>N                        | <i>)</i> |   |    |                          |                   | 子の一般の一個の一個の |    |   |   |   |                   |                      |                      |   | Reque  |   |
| Con                           |          |   |    |                          |                   | Com         |    |   |   |   |                   |                      |                      |   | Requested Analysis   |   |
| Samples Intact/ Y) or         |          |   |    |                          |                   | Comments    |    |   |   |   |                   |                      |                      |   |  |   |
| Y)<br>Pr                      | Y        |   |    |                          |                   |             |    |   |   |   |                   | LAB USE ONLY         |                      |   |  |   |

<sup>\*\*\*</sup>In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.

# Pace Analytical\*

# Document Name: Sample Condition Upon Receipt Form

Document No.: F-DUL-C-001-Rev.01

Document Revised: 22Jan2016 Page 1 of 1

Issuing Authority: Pace Virginia, Minnesota Quality Office

| Sample Condition   Client Name:   Upon Receipt     COC Virgi                                    | n,<br>n, a     |                     | Project #    | :   |
|---|----------------|---------------------|--------------|---|
| Courier: Fed Ex DUPS  | USPS Other:    | <b>7</b> 0/40<br>□□ | ient<br>し    |   |
| Tracking Number:  |                |                     |              |   |
| Custody Seal on Cooler/Box Present? Yes   | No             | Seals In            | tact?        | Yes No Optional: Proj. Due Date: Proj. Name:  |
| Packing Material: Bubble Wrap Bubble Bag  | s 🔲 No         | ne 🗌                | ]Other:      | Temp Blank? Yes No  |
| Thermometer Used: B00051  | Type of lo     | :e: [/]             | Wet [        | Blue None Samples on ice, cooling process has begun                                   |
| Cooler Temp Read °C: 0 - 5 Cooler Temp Co   |                |                     |              | Biological Tissue Frozen? Yes No NA Initials of Person Examining Contents:  Comments: |
| Chain of Custody Present?   | Yes            | □No                 | □n/A         | 1.  |
| Chain of Custody Filled Out?  | ੋ <b>ਪ</b> Yes | □No                 | □n/a         | 2.  |
| Chain of Custody Relinquished?  | ¥es            | □No                 | □n/a         | 3.  |
| Sampler Name and Signature on COC?  | Yes            | □No                 | ™N/A         | 4.  |
| Samples Arrived within Hold Time?   | ا Yes الط      | □No                 | □N/A         | S   |
| Short Hold Time Analysis (<72 hr)?  | ∐Yes √         | V No                | □N/A         | 6.  |
| Rush Turn Around Time Requested?  | ∐Yes           | N0                  | □N/A         | 7.  |
| Sufficient Volume?  | Yes            | □No                 | □N/A         | 8.  |
| Correct Containers Used?  | Yes            | □No                 | □N/A         | 9.  |
| -Pace Containers Used?  | Yes            | □No                 | □N/A         |   |
| Containers Intact?  | Yes            | □No                 | □N/A         | 10.   |
| Filtered Volume Received for Dissolved Tests?   | ∐Yes           | □No                 | N/A          | 11. Note if sediment is visible in the dissolved containers.                          |
| Sample Labels Match COC?  | Yes            | □No                 | □n/a         | 12.   |
| -Includes Date/Time/ID/Analysis Matrix: WT  |                |                     |              |   |
| All containers needing acid/base preservation will be checked and documented in the pH logbook. | ∐Yes           | □No                 | ∏n/a         | See pH log for results and additional preservation documentation                      |
| Headspace in Methyl Mercury Container   | Yes            | □No                 | □N/A         | 13.   |
| Headspace in VOA Vials ( >6mm)?   | Yes            | □No                 | □N/A         | 14.   |
| Trip Blank Present?  Trip Blank Custody Seals Present?  | ∏Yes<br>∏Yes   | □No<br>□No          | ∏N/A<br>∏N/A | 15.   |
| Pace Trip Blank Lot # (if purchased):   | ∐1es           |                     | ΨМ           |   |
| CLIENT NOTIFICATION/RESOLUTION  |                |                     |              | Field Data Required? Yes No   |
| Person Contacted:   |                |                     |              | Date/Time:  |
| Comments/Resolution:  |                |                     |              |   |
|   |                | **                  |              |   |
|   |                |                     |              |   |
| FECAL WAIVER ON FILE Y N  | - In           | TEM                 | IPERATU      | IRE WAIVER ON FILE Y N  7-18-16   |

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)